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by

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THE METHOD OF INVESTIGATING METEORITES AND TEKTITES WITH THE AID OF INFRARED SPECTROMETRY[1]

by A. A. Kadushin
and G. G. Vorobyev

The emission method of spectral analysis, widely and successfully used in meteoritics [1], [2], is based on the electron transition of the atoms of chemical elements. The radiation of light energy related to such transition makes it possible to fix that energy at certain frequencies of the ultraviolet and visible spectrum. The infrared region imposes certain limitations on the emission analysis [1], but opens wide possibilities for a younger field of investigation, absorption (molecular) spectrometry.

Most of the oscillation and rotation frequencies of atoms and radicals are found in the infrared region in the form of absorption bands which vary not only in position but also in structure. Consequently, if the emission analysis provides an idea of the elementary composition of matter and makes a deduction of its empirical formula possible, an absorption analysis interprets molecular structure, defines the separate functional groups and fixes the chemical bonds (such as C-H, O-H, N-H, Si-O, etc.).

The gist of the method is that the thermal rays from the infrared radiation source (a pin made of rare earth or carborundum oxides and heated with an electric current), passing through a layer of the matter under consideration (in a solid, liquid or gaseous state), is decomposed with the aid of a diffraction grating or optical prisms (of NaCl, KCl,

1. The authors are grateful to A. V. Kokhmanskiy of the All-Union Scientific-Research Institute of NSDV for his technical assistance in this work.

KBr, CsBr, KJ, LiF or others); this causes the absorption of these rays in certain sections of the spectrum, in accordance with the molecular composition of the matter.

Many years have gone by since 1881 when Abney and Festing first proposed their hypothesis of a qualitative and quantitative bond between the atoms in a molecule and the infrared absorption spectra before that method was accorded due recognition and adopted for practical development which has been hindered primarily by instrumentation difficulties. Coblenz (1905) was among the first to point to its importance for physicochemical analysis. By 1938 the number of infrared installations in the U.S., where that method was under development, was 15, but at the end of November 1947 there were already more than 500 of them, and their number continues to grow [3].

Infrared spectrometry is currently widely used in the petroleum and chemical industries (mostly for analyzing liquid organic products). In 1950, Hunt, Wisherd and Bonham [4] investigated the spectra of over 100 inorganic compounds, pure salts and minerals, and showed that solid substances produce simpler and fewer absorption bands as compared to liquid substances, and that they can also be used for identification purposes. The infrared method has only recently [5-7] been introduced in mineralogy, and the nature of its application is still cautious and selective. Under study now are mostly argillaceous minerals as well as some relatively simple classes of compounds: carbonates, sulfates and phosphates.

Meteorites have a specific and relatively uniform mineralogical composition which considerably facilitates their investigation by the infrared method. However, the complete insensitivity of this method to native elements restricts its application only to stony meteorites which consist

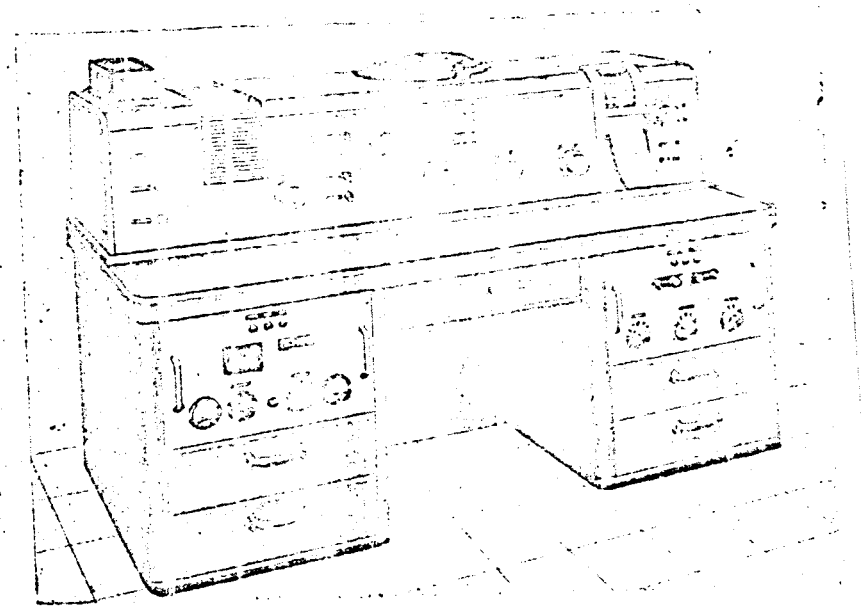

Fig. 1. The UR-10 automatic infrared spectrometer (made by the Karl Zeiss Co.)

primarily of silicates (olivine, pyroxene, plagioclase) and sulfides (troilite). As for the small residual inclusions of nickelous iron in the almost pure non-magnetic fraction of the meteorites, they are automatically excluded from the analysis, and the resulting spectrum represents an absolutely pure silicate-sulfide phase. This circumstance is a matter of definite interest to meteoritics whose chief problem is to classify the minerals according to their magnetic properties.

From a mineralogical point of view, the tektites represent well-fused silicate glass. As amorphous bodies, they make the application of x-ray and differential thermal methods of analysis impossible. The infrared method is therefore practically the only remaining structural method of investigation. The first publications of the tektite spectra [8] and [9] were of an exploratory nature; they have attracted the attention of the researchers and called for a continued effort in that direction.

The apparatuses still produced by the industry are designed for a

relatively narrow spectral region from 5 to 15 μ. An extension beyond that region required the use of costly prisms, the production of which was not organized. The change of the prisms involved a great deal of time which made it impossible to obtain a continuous spectrum.

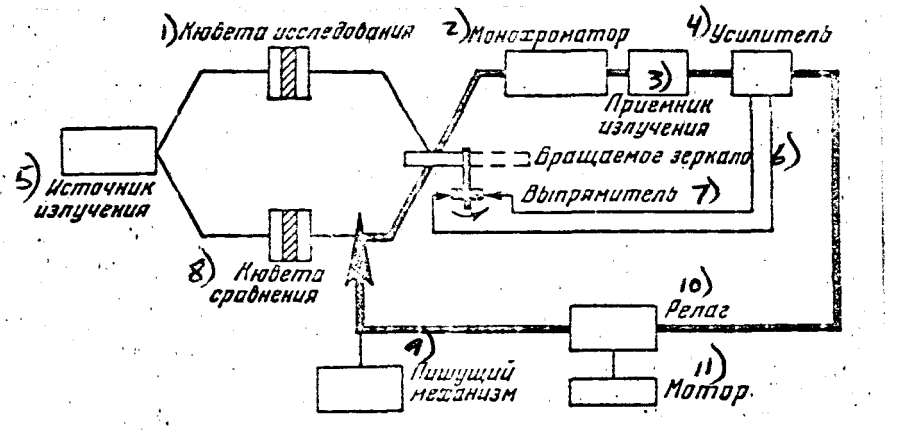

Fig. 2. Basic diagram of the UR-10 infrared spectrometer.

Legend:
1-investigation cuvette
2-monochromator
3-radiation receiver
4-amplifier
5-radiation source
6-rotatable mirror
7-rectifier
8-comparison cuvette
9-writing mechanism
10-"relag" [relaxation oscillator?]
11-motor

The Karl Zeiss Company of Jena, East Germany, is now producing a UR-10 spectrometer (Fig. 1) designed to work within the range of 2-25 μ (5000-400 cm^{-1}) by way of the automatic change of the following three prisms: KBr, NaCl and FiF. The basic diagram of this apparatus is shown in Fig. 2. The radiation from the source passes simultaneously through the substance under analysis and the standard (control) substance and hits a mirror rotating at a frequency of 10 cps. The result is an alternate projection of the rays from two channels to the inlet aperture of the monochromator. Decomposed in the latter, the rays pass through the outlet aperture to the receiver. If the radiation on both channels is attenuated unevenly, the alternating voltage coming from the receiver is amplified by an electronic amplifier and fed to the servomotor through a

phase detector synchronized with the modulating mirror. A special diaphragm introduced with the aid of that motor equalizes the radiation of both channels reducing the alternating component to zero. As the diaphragm is shifted, the substance passing through the given region of the spectrum is recorded.

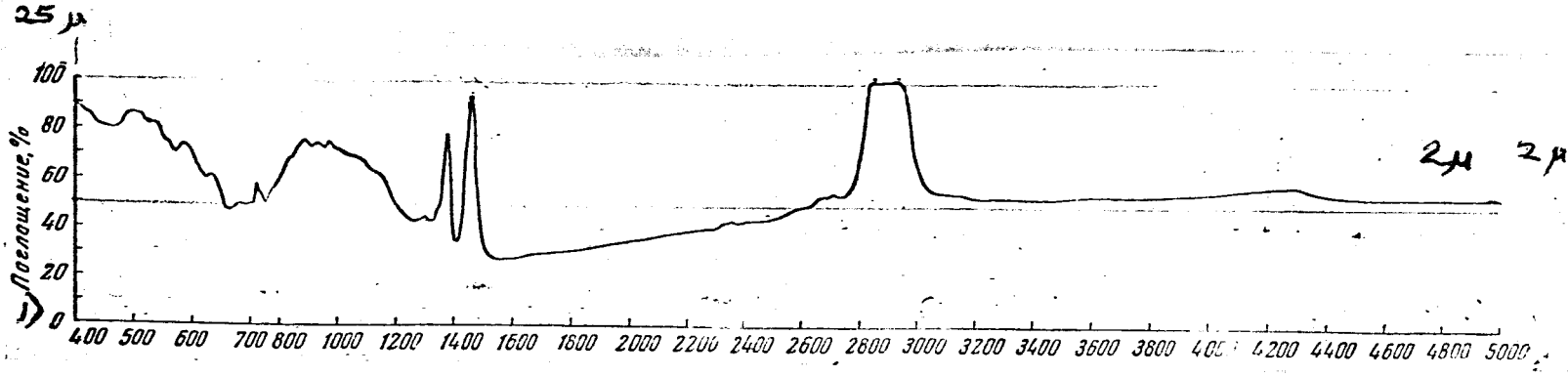

Fig. 3. The absorption spectrum of the Nikol'skoye meteorite recorded by the suspension method.

Legend: 1) absorption.

The advantage of the double-beam method of alternating radiation (as compared to the more popular single-beam method) is that the recorded spectra are completely free from the vapor-absorption bands of the atmospheric water and carbon dioxide. The fluctuations of the radiation source, the sensitivity of the receiver and degree of amplification do not affect the registration quality. The registration speed can be changed from 4 to 400 cm^{-1}/min. A special device makes it possible to reduce the registration speed for a more accurate recording of the structure of the individual bands. The wavelength scale in the apparatus is almost linear within the entire range, and is recorded directly on the registration paper which prevents possible errors due to slippage or the improper insertion of the tape with a pre-recorded coordinate grid. The average reproducibility of the number of waves is at least 1 cm^{-1}, and the reproducibility of the passage is less than $\pm$0.5%. The resolving power of the

instrument is about 2 cm^{-1}.

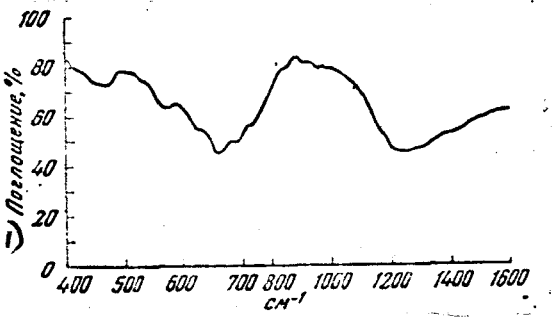

Fig. 4. The absorption spectrum of the Nikol'skoye meteorite recorded by the powder method.

Legend: 1) Absorption

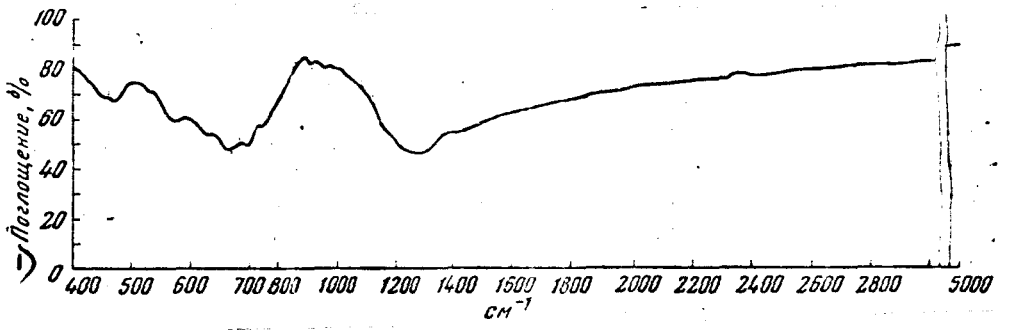

Fig. 5. The absorption spectrum of the Noyan-Bogdo meteorite recorded by the powder method.

Legend: 1) Absorption

The limited experience in the use of this apparatus applies primarily to liquid, gaseous and to some extent solid organic substances introduced in special cuvettes. An analysis of the meteorites and tektites calls for a new complex of preparatory operations since the substances are insoluble in ordinary solvents, and are fusible only at high temperatures. The following three methods were tested within the framework of this experiment: 1) suspension, 2) powder and 3) tablets. The first method is used to produce a suspension of a finely pulverized substance in some liquid (usually in hydrocarbon oil). A thin layer of the prepared paste is spread on a NaCl (or KBr) plate and compressed by another plate. Naturally, the spectrum produced by this method is a combination of two spectra,

of the substance under analysis and the oil. The second method consists in the suspension of the substance in a volatile liquid (such as CS_2 or CCl_4). When the suspension is spread on a plate, the liquid evaporates and the remaining layer of powder of uniform thickness is compressed by another plate. The best method for a quantitative analysis is to table the powder in a mixture with KBr or NaCl.

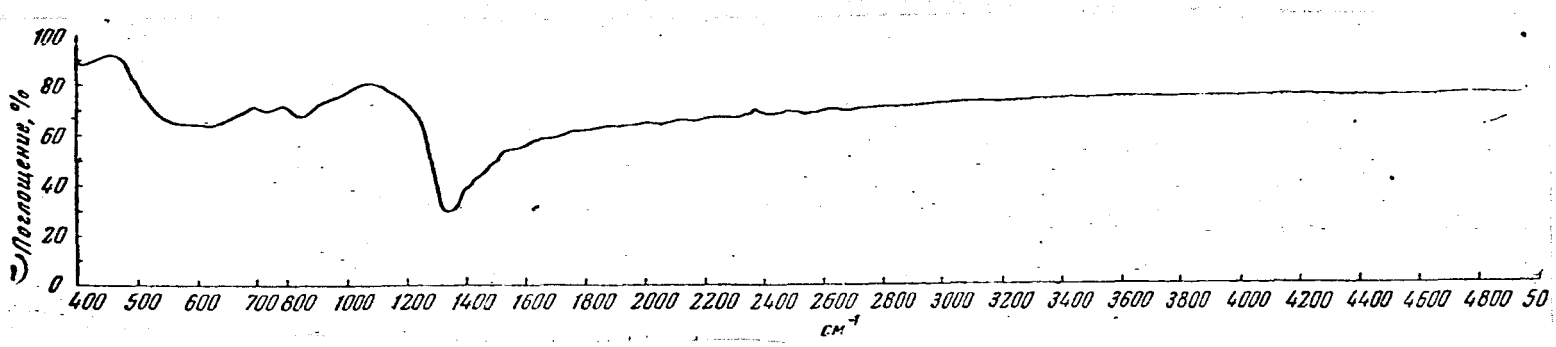

Fig. 6. The spectrum of a moldavite-tektite (from Radomilitsy, Czechoslovakia) recorded by the powder method.

Legend: 1) Absorption.

It has been established that within the range of wavelength above 2 μ, the diffusion phenomenon can distort the resulting spectrum to a considerable extent. That phenomenon is due to the fact that the diffusion produced by the solid particles in the infrared rays is greater than the absorption, and the maximum magnitudes of the bands are undergoing arbitrary changes. Consequently, when preparing the powder, a maximum and constant degree of pulverization should be achieved, with the average size of the particles not exceeding 5 μ [7], [10] and [11]. A vigorous grinding of rock crystal in a jasper mortar for 5, 10, 15, 30 minutes and one and two hours showed that 15-30 minutes was the optimum duration.

The absorption spectra of the non-magnetic fraction of various meteorites as well as tektites and other natural glass were obtained by the above-described method. A preliminary examination of the spectrum of the

Nikol'skoye meteorite by the method of suspension in hydrocarbon oil revealed (Fig. 3) that the absorption bands of the meteoritic minerals are found in the range of 6μ and higher. The oil absorption bands are found in the same range: about 14μ of the CH_2 groups, about 7μ of the CH_2 and CH_3 groups, and the wider bands of C-H oscillations are located in the region of 3-4μ. Thus the use of oil for analyzing meteoritic matter has been found to be impractical.

Fig. 4 shows the spectrum of the same meteorite recorded by the powder method within the range of 6-25μ. It is identical with the previous spectrum and is free from the absorption bands of the hydrocarbon oil. The powder methods was also used to record the spectrum of another meteorite, the Noyan-Bogdo (Fig. 5). These two chondrite-meteorites have a somewhat similar mineralogical composition, which is reflected in the nature of the absorption curves. A slower scanning of the spectrum within a narrow range of wavelengths reveals individual structural details of the bands in connection with the changing composition of the individual minerals and the relationship between them. The moldavite-tektite (Fig. 6) has an entirely different type of spectrum reflecting the composition and structure of silicate glass, an amorphous silicon dioxide with oxides of other elements dissolved in it.

The results of all these data and the identification of the individual bands according to their chemico-mineralogical composition will be dealt with in a separate article.

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